

1 Title: CONTROLLED LOW STRENGTH FLOWABLE FILL COMPOSITION
2 WITH IRON CHELATING COMPOUNDS
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4 This application claims priority to co-pending U.S. Provisional Application
5 Serial No. 60/453,231 filed March 10, 2003.

6 Background of the Invention

7 This invention relates to Controlled Low-Strength Mixtures (CLSMs), or
8 flowable back-fills. This class of materials has utility as pipe bedding materials has
9 utility as pipe bedding materials where they are used to both protect the pipe from
10 external agents and internal loads. They have also been used as an erosion barrier in
11 embankments and as a mine fill material. CLSMs typically have strengths of less than
12 2000 psi and, in cases where removal is contemplated, less than 200 psi for ease of
13 removal. The material should be initially in the form of an easily pumpable, self-
14 leveling slurry. Rapid early strength development (approximately 50-70 psi) is a
15 desirable property and is currently not obtainable with commercial products without
16 the penalty of high strength development at later stages. U.S. Patent No. 5,106,422
17 discloses Class C Fly ash in a rapid setting flowable backfill composition and method
18 for its use.

19 However, such existing compositions are based upon the use of either Portland
20 cement or Class C fly ash used individually or in combination as the hydraulic cement
21 component of the CLSM system. Typically these cementitious materials are used at
22 less than 5% by weight in the case of Portland cement or as much as 50% in the case
23 of Class C fly ash with the remainder being some form of aggregate, usually fine sand
24 or soil from the spoil with small amounts of additional rock and gravel or Class F fly
25 ash. Cement-based materials can take days to hydrate, cure, and achieve even a

1 modest strength of 50 psi which is typically the minimum strength required for a man
2 to walk upon the surface of the bedding material and represents the minimum safe
3 time before the cover fill may be placed. Class C fly ash based systems may take as
4 long as four hours to hydrate, cure, and achieve this strength. In many cases, locally
5 available Class C fly ash is not desirable for use in these types of product due to slow
6 hydration, cure, and set times and low strengths. Strength may be compensated for by
7 the use of additional Class C fly ash but the cost of the additional fly ash may result in
8 cost prohibitive products.

9 Thus, the system of the present invention minimizes the down time before
10 cover fill may be placed and represents a significant savings of both time and money
11 for the user. Furthermore, the present inventive composition and method allow for
12 control variability in strength and hydration, cure, and set times of a CLSM system
13 utilizing Class C fly which results in a Class C-based flowable fill capable of
14 competing in markets previously inaccessible.

15 BRIEF DESCRIPTION OF THE DRAWINGS

16 Fig. 1 is a graphic representation of the effect of set time of Class C mortars
17 with lime.

18 DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

19 Class C fly ash as defined in ASTM C 618 is a coal combustion product that
20 meets particular size requirements and mineralogical specifications. A typical
21 chemical composition for this class of fly ash is as follows:

22

	<u>Percent by Weight</u>	
2	Silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3), min.	50.0
3	Sulfur trioxide (SO_3), max.	5.0
4	Moisture content, max.	3.0
5	Loss on ignition, max.	6.0

7
8 This is a rather broad description for this class of material, and significant
9 variability may exist for materials conforming to this requirement. The variability
10 manifests itself as differences in hydration and set time and strength between several
11 samples of Class C fly ash either from the same or different sources. It has been found
12 that a major factor contributing to variability is the amount of available calcium
13 present in the sample. Additionally, soluble iron content contributes to slow setting
14 times. Furthermore, the addition of small amounts of calcium to Class C fly ash has
15 no deleterious effects upon flowable fill and can accelerate the rate of hydration and
16 cure while minimizing the differences in set time and strength of flowable fill mixtures
17 containing Class C fly ash.

18 Where soluble iron is present in sufficient quantity and extra calcium alone is
19 inadequate to accelerate the rate of hydration, iron chelating compounds may be
20 added, even in very small amounts, to offset the soluble iron effect. The iron chelating
21 compound may include:

22 sodium chloride, sodium thiosulfate, triethanolamine, diethanolamine,
23 polyethyleneimine, amino-substituted acrylic monomers or polymers,
24 morpholine and substituted morpholine compounds, urea, guanidine salts,
25 pyrole and pyrole compounds, polyvinylpyrole, imidazole compounds,
26 pyrazoles, pyridine and pyridine compounds (especially ortho alkoxy-

1 substituted pyridines), amino phenol (especially ortho amino phenol), amino
2 cresol, ortho anisidine, amine acetate surfactants (such as Armac HT and
3 Armac 18D-40 from Akzo Nobel Chemicals), amine oxide surfactants (such as
4 Ammonyx series of surfactants from Stepan Company, Schercamox series of
5 surfactants from Scher Chemicals, Foamox series of surfactants from Alzo,
6 Inc., Chemoxide series of surfactants from Chemron Corp.), amine surfactants
7 (such as the Armeen and Redicote series of surfactants from Akzo Nobel
8 Chemicals, the Incromine series of surfactants from Croda, Inc., the Tealan
9 series of surfactants from R.I.T.A. Corp.), and mercapto surfactants (such as
10 Burco TME from Burlington Chemicals).

11 The iron chelating compound may be in quantities in the range of 0.01% or
12 5.0% by weight. Effective results have been obtained and reasonably should be
13 obtained from chelting agents or compounds selected from the group consisting of an
14 alkanolamine, a polymer of ethyleneimine, a block copolymer containing
15 polyethyleneimine segments, an amino-substituted polymer of acrylic acid, the salt of
16 an amino-substituted polymer of acrylic acid, a carboxyated amine compound, a salt
17 of a carboxyated amine compound, ethylenediaminetetraacetic acid and salts thereof;
18 nitrilotriacetic acid and salts thereof, an amine substituted surfactant, an amine oxide
19 substituted surfactant, and a guanidine salt.

20 The following examples illustrate the nature of the present invention. Set
21 times were determined when a 0.25" diameter penetrometer needle provided a reading
22 of 200 psi on insertion to a depth of 1.0".

1

2 Example 1

3 Coal Fly ash from Deeley Power Plant, San Antonio, Texas, as obtained and
4 used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed
5 silica sand (Espey Sand, San Antonio, Texas) and 35 mL deionized water were mixed
6 for 1 minute and poured into a 2" cube mold. The set time was determined to be 62
7 minutes as shown in Table 1 below.

8 Examples 2-8

9 Coal Fly ash from Deeley Power Plant, San Antonio, Texas, was obtained and
10 used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed
11 silica sand (Espey Sand, San Antonio, Texas), varying amounts of type S hydrated
12 lime and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube
13 mold. The set time for these examples are shown in Table 1 for the varying amounts
14 of lime.

15 Examples 9-13

16 Coal Fly ash from Scherer Power Plant, Atlanta, Georgia, was obtained and
17 used as received. 50 grams of Class C Fly ash, 250 grams ASTM C 33 graded washed
18 silica sand (Espey Sand, San Antonio, Texas), varying amounts of type S hydrated
19 lime and 35 mL deionized water were mixed for 1 minute and poured into a 2" cube
20 mold. The set times for these examples are shown in Table 1 for the varying amounts
21 of lime.

1 A graphic representation of the effect of set time of these Class C mortars with
 2 lime of varying amounts is shown in Fig. 1.

3 Table 1. Set times of Class C Fly ash mortar cubes containing varying amounts
 4 of type S lime.

Lime (grams)	Example Number	Set Time (minutes) Deeley	Example Number	Set Time (minutes) Scherer
0.00	1	62	9	348
0.07	2	51	-	-
0.13	3	26	-	-
0.25	4	12	10	303
0.50	5	10	11	71
0.75	6	8	12	37
1.00	7	12	13	76
1.50	8	9		

5
 6
 7
 8 Example 14
 9

Ingredient	Amount (g)
Concrete Sand	0
Type C Fly Ash	100
Hydrated Lime	.003
Triethanolamine	.04
Water	25

10
 11 The dry ingredients were mixed together and the water was added with mixing
 12 until a smooth, pourable consistency was obtained. The set time was 17 minutes.
 13
 14

15 Example 15
 16

Ingredient	Amount (g)
Concrete Sand	200
Type C Fly Ash	100
Hydrated Lime	.3
Triethanolamine	.48
Water	39

17

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2 The dry ingredients were mixed together and the water was added with mixing
3 until a pourable consistency was obtained. The set time was 17 minutes.

4

5

6 Example 16

7

Ingredient	Amount (g)
Concrete Sand	250
Type C Fly Ash	50
Hydrated Lime	7.5
Triethanolamine	.1
Water	40

8

9 The dry ingredients were mixed together briefly and the water and
10 triethanolamine added with continued mixing. The set time was 23 minutes.

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